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FAT AND MOISTURE TESTING METHODS

GOVERNMENT REGULATIONS of meat composition and labeling plus the higher price of meat protein has made analysis for fat, moisture and protein increasingly important to the meat processor.

Production time considerations and the widespread nature of the problem demands an analysis system that is fast and inexpensive enough for on-line testing by even the small processor. The official AOAC methods are inexpensive and accurate but much too slow—a minimum 1 hour for fat and 2 hours for moisture.

The following survey of currently available methods for analyzing meat is based on research by J. D. Pettinati, C. E. Swift and E. H. Cohen of the USDA's Agricultural Research Service, Eastern Regional Research Center, Philadelphia. They are currently attempting to find practical alternatives to the official testing methods. Further information was gathered directly by WESTERN MEAT INDUSTRY.

The table compares the methods studied with the accuracy of the AOAC methods. Some newer equipment still has not been studied so the results of the research are still somewhat tentative and arbitrary.

Chosing the proper testing equipment for a specific processing operation should be based on the right combination of equipment characteristics. These variables include: the size of the sample, whether the sample must be fresh or can contain additives, whether the sample is usable after the test, the cost of the equipment and the technical skill necessary for the operation.

Of course, the accuracy of the method is of prime importance. The USDA researchers consider a standard deviation below 0.7 to be quite good. The AOAC methods in comparison have standard deviations of plus or minus 0.29% for moisture and plus or minus 0.40% for fat.

A large size sample yields better results for ground meat with large meat particles since sampling and mixing become less rigorous. For emulsions the sample size is not as important but the method must not be sensitive to non-meat additions if the method is to be used on the final product.

The table comparing the analytical methods was published in the Journal of the Assoc. of Official Analytical Chemists in May, 1973. Therefore, the prices indicated may not be current in some cases.

The mean difference indicated in the table is the average algebraic sum of differences in the percentage fat or moisture between the stated method and the official AOAC method. The range of differences shows the results of ex-

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	Method[a] No.	Type	Essential characteristic	Time required for a single analysis	Sample size,[b] g	No. of analyses[c]	Mean difference,[d] % fat	Range,[e] % fat	Std dev.,[f] % fat	Cost of equipment[g] and training[h]
Solvent Extraction	1	AOAC official	Soxhlet or Goldfisch extraction	7 hr	3–4	240	0	±0.7	0.25	$75 per Soxhlet unit; $650 per Goldfisch unit, 2
	2	Modified Method No. 1	rapid 125° drying and rapid extraction	¾–1¼ hr	3–4	27	0	−0.8 to +0.2	0.6	same as above
	3	Moisture balance (Ultra-X)	CCl_4 extraction after drying	35–55 min	2.5	23	−0.1	−2.4 to +1.8	1.0	$1250, 1
	4	Extracted residue (Si-Mo-Fat)	micro vacuum oven	1–2 hr	10	46	−1.0	−2.6 to +0.7	0.8	>$200, 2
	5	Distillation extract residue (Cohen)	azeotropic distillation-extraction, evaporation	¾–1 hr (for both moisture and fat)	10	16 16	−0.7 (*m*-xylene) −0.3 (cumene)	±0.9 ±0.5	0.4 0.5	$75, 2
	6	Chloroform-methanol solvent extraction	gravimetric	10 min	100	28	0.3	not available	0.2	$100.2
Babcock	7	Modified Babcock	modified Paley bottle, with or without centrifugation	20–30 min	9	240 18	−0.1 0.2	±1.0 −0.6 to +1.2	0.3 0.4	<$50 (for 6 samples), 1
	8	Modified Babcock, $HClO_4$-HOAc	same as above	30 min	9	19	0	−0.4 to +0.5	0.2	same as above
	9	Modified Babcock, HCl-DMSO	same as above	15 min	9	98	−0.2	−1.3 to +1.6	0.6	same as above
	10	Modified Babcock (Banco)	same as above, Banco reagents	35 min	9	32	0.2	−0.8 to +1.2	0.4	$300, with centrifuge, 2
Rendering	11	Rendering (Hobart)	inverted hot plate and collector	15 min	56.6 (2 oz)	64	−2.3	−4.1 to −0.5	not available	$195, 1
	12	Rendering (Goss)	resistance heater and collector	15 min	15–25	not available	not available	±1.5	not available	<$100, 1
	13	Microwave oven (Hobart)		4 min	70	4	0.3	-0.2 to 1.09	0.6	$5000.2
Specific Gravity	14	Specific gravity (Honeywell)	semiautomatic weight and volume	3 min	750	69 56	0.1 −1.9	±2.4 ±3.4	1.2 1.5	$10,000 or rental, 1
	15	Specific gravity of extracts	heptane extracts, hydrometers	15 min	20–40	17	0.3	−0.9 to +0.8	0.5	>$300, 2
	16	Specific gravity of extract (Foss)	Tetrachloroethylene extraction	4–5 min	45	68		3.1 to 2.1	0.7	$4200.2
Other Methods	17	Separatory funnel	extraction 5× with ether	1.5–2 hr	5	23	0.2	−1.1 to +1.5	0.7	<$50, 1
	18	Infrared absorption	spectrophotometry	<5 min	1	34	not available	not available	1.4	>$3,000, 3
	19	X-ray absorption (Anyl-Ray)	electronic unit	5 min	5902 (13 lb)	12	not available	±1.5	0.5	$5,000/year rental, 1
	20	Capacitance (Steinlite)	solvent extraction	20 min	50	20	9.2	−1.9 to +1.2	0.8	>$500, 2
	21	Capacitance (AMIF)	distillation extract is measured	30 min	15	19	0.3	−2.1 to +2.9	1.3	>$500, 2
	22	Infrared absorption (Neotec)		10 sec	450			-1 to 1	0.8	$350/yr. rental
	23	Electrical conductivity (Emme)		10 sec	60 lb.	30		-2.2 to 3.3	1.54	$28,000.1

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	Method[a] No.	Type	Essential characteristic	Time required for a single analysis	Sample size,[b] g	No. of analyses[c]	Mean difference,[d] % moisture	Range,[e] % moisture	Std dev.,[f] % moisture	Cost of equipment[g] and training[h]
Evaporation	24	AOAC official	100–102° air oven	16–18 hr	4–6	108	0	not available	0.25	>$250, 1
	25	AOAC official	125° mechanical convection oven	2–4 hr	4–6	108	0.2	−1.0 to +1.8	0.33	>$500, 1
	26	Modified Method No. 2	125–150° gravity oven	2–4 hr	4–6	12	0	−0.3 to +1.0	0.7	>$250, 1
	27	High temperature oven	200° mechanical convection oven	>15 min	25	25	0	−0.4 to +0.5	0.2	>$500, 1
	28	AOAC official	95–100° vacuum oven	5 hr	4–6	72	0.2	−1.5 to +1.1	0.4	>$150, 1
	29	Low temperature vacuum oven	70° vacuum oven	16–18 hr	4–6	101	−0.1	−1.4 to +3.4	0.6	>$150, 1
	30	Hot plate	200° at surface	30–45 min	10	24	0.7	−0.9 to +0.7	0.7	<$100, 1
Infrared Absorption	31	Infrared radiation oven (Dynatronic)	IR heater, built-in balance	40–60 min	12	24	0.1	±0.6	0.5	>$600, 1
	32	Infrared radiation lamp (Ultra-X)	IR lamp, built-in balance	15–25 min	2.5	23	−0.5	−1.7 to +2.2	1.0	>$400, 1
	33	Infrared balance (Ohaus)	IR lamp, separate balance	30–45 min	10–20	24	0.4	−0.3 to +0.4	0.3	$100, 1
Azeotropic Distillation	34	Azeotropic distillation (Si-Mo-Fat)	distillation with butyl ether	2 hr	10	46	1.0	−0.7 to +2.3	0.8	>$200, 2
	35	Azeotropic distillation (AMIF)	distillation with 2-octanol	15 min	15	19	−0.7	±1.9	1.1	>$100, 2
	36	Azeotropic distillation (Cohen)	distillation with various solvents	15 min 30 min	10 10	24 24	0	−0.7 to +1.3	0.5	<$100, 2
Other Methods	37	Refractometry	methanol extract	5–10 min	20	36	-0.5	-0.9 to 0.16	0.3	$650.2
	38	Gas-liquid chromatography	methanol extract	10 min	2	74	0.3	-1.4 to 1.5	0.3	$18,000.2

The above tables were published in the Journal of the Association of Official Analytical Chemists (Vol. 56, No. 3, 1973). Additional methods (No. 8, 13, 16, 35 and 36) were obtained from a subsequent paper by Pettinati, Swift and Cohen presented at the 26th Annual Reciprocal Meat Conference of the American Meat Science Assoc. Data for methods No. 22 and 23 were obtained directly from the manufacturers.

periments after the mean difference was added or substracted. The training necessary is indicated in the last column by the number 1 for brief training; 2 for moderate; and 3 for thorough training.

FAT TESTING

SOLVENT EXTRACTION

As with most analytical methods that yield high accuracy, the official AOAC method of fat analysis is very time consuming. The ether extraction of fat from the meat product and its subsequent distillation requires 7 hours (See table, method No. 1). A modification (method 2) of this still takes ¾-1¼ hrs. This amount of time precludes using this method for such control measures as preblend analysis.

The solvent extracted from the residue of azeotropic distillation for moisture testing (methods 32, 33, and 34 in the table) can be used to determine fat (methods 4, 5 and 21). The Ultra-X (method 30) has a dry residue suitable for solvent extraction for fat.

Most of the solvent extraction methods are quite lengthly except for the chloroform-methanol method (No. 6) which requires only 10 minutes. The accuracy was very good but the USDA researchers warn that the results came from tests on only low fat meat types—lamb and veal. Extraction with m-xylene or cumene (method 5) is only fast enough for screening but the method is accurate and thoroughly tested.

The equipment involved in these extractions are not expensive and the training is moderate.

BABCOCK TYPE METHODS

The Babcock type methods are relatively rapid (8-30 min.), are relatively easy to perform and require a low investment in equipment. Acid or enzyme is used to digest the meat and then a centrifuge separates the fat so it can be measured by volume. No spices can be in the tested meat product since they will float below the fat rendering the measurement inaccurate.

The use of sulfuric acid for digestion is a problem in some of the methods since it is hazardous for the technician and it may char some samples. The type of acid to be used depends on the type of meat product to be tested.

Digestion by heating sulfuric acid and hydrogen peroxide is claimed to have a very broad range of applicable meat products but the method is not yet thoroughly tested.

RENDERING METHODS

Simple heating of the sample until it melts into a collecting vessel is the basis for the rendering method. The rendered fat is measured by volume or gravimetrically. This measurement would be more direct if the fat were pressed out of the residue. These methods are fast (4-15 minutes) and quite accurate.

The microwave system (method 13) is relatively new but if it proves as accurate as the preliminary tests indicate it would be very useful since it requires only four minutes. After the moisture is vaporized and the fat is melted, the residue from this method can be used to test for protein and ash.

SPECIFIC GRAVITY

The Honeywell (No. 14) and Foss (No. 16) systems are very attractive especially for processing screening due to their speed.

The Honeywell system requires a large sample but the meat is recoverable. The system must be programmed for use with each different type of fresh meat.

The Foss system takes slightly longer and renders the sample unusable but it has demonstrated higher accuracy on fresh meat.

OTHER INSTRUMENTS

X-ray transmission, electrical conductance, infrared reflectance and magnetic resonance have also been used for fat analysis instruments.

X-rays are used in the Anyl-Ray system (method 19) to measure that amount that will pass through a path in the meat. The samples are 13 lbs. but they are reusable. The meat does not have to be ground but it must be properly compacted, and free of salt and some other additives. Samples smaller than 13 lbs. can also be used if they are thoroughly mixed and very uniform.

Measurement of the absorption by meat of infrared light waves yields very rapid and accurate results. The accuracy depends on the size to which the meat is ground. Substances other than meat interfere with the measurement so it must be used for fresh product.

A ground meat analyzer by Neotec (method 22) is inexpensive, requires only ten seconds and can take the measurement when the ground meat is already in a clear plastic display container. Its accuracy is still being studied by the USDA but it will certainly prove useful at least to the retail stores.

A more sophistocated and more expensive system, under development by Computer Concepts, determines the fat, protein and moisture content of an emulsion smeared over a glass viewing plate. The three readings are delivered in ten seconds in a digital readout display.

Non-specific spectrophotometric equipment costs over $3000 and would require a skilled technician. Several companies have used this same principle in equipment that is more convenient to use.

The capacitance measurement devices, Steinlite and AMIF, (methods 20 & 21) are sensitive to variations in temperature.

Using the fact that lean meat conducts electricity about 20 times better than fat, the Emme ground meat analyzer (method 23) measures the total leanness of fresh meat.

Measurement of an entire 60 lb. box of meat requires only 10 sec. Only fresh meat can be tested.

Bruker Scientific has an instrument which measures both fat and moisture by means of nuclear magnetic resonance. The system is fast (10 sec.—5 min.) but it can only accept a sample measuring one cubic centimeter and it costs about $12,000.

A wet chemistry fat testing system is under development by Technicon.

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Protein content is usually found by substracting the fat and moisture from the total weight. A rapid method has been developed by Baltimore Spice Co. for measuring protein directly. The instrument is based on dying of the protein and measuring the amount of protein-binding dye which has not been bound by the protein.

About 15 min. are required for the test on a 40 gm sample. No special technical skill is needed to operate the $1100 instrument. Standard deviate from the official Kjeldahl method are about 0.2.

MOISTURE TESTING

OFFICIAL AOAC METHOD

Testing for moisture is more difficult than might be expected since water is bound both chemically and physically in the meat. In heating, accurate measurement is further complicated by the hardening of the surface which makes evaporation from the interior more difficult. Raising the temperature will lead to inaccurate results due to fat spattering, sample decomposition and oxidation.

The official AOAC method (No. 22, 23, 24 & 26) involves heating in an air oven at 100-102° for 16-18 hr., or in a mechanical convection oven at 125° for 2-4 hr., or in a vacuum oven at 95-100° for five hr. After cooling, the weight of the sample is compared with its original weight. Though these methods are very accurate, simple and inexpensive, they are quite time consuming. Hot plate methods are faster but accuracy suffers somewhat.

Alternatives to slow heating in ovens include titration, azeotropic distillation, infrared radiation, microwave adsorption, infrared reflection, refractometry and gas-liquid chromotography.

INFRARED RADIATION

Moisture balances (methods 29, 30 & 31) reduce the time necessary for evaporation of the moisture to less than one hour and in one case down to 15 min. Infrared lamps heat the sample while it is weighed for weight loss on a scale. The measurements deviate very little from the official methods.

AZEOTROPIC DISTILLATION

Methods (No. 32, 33 & 34) $2000. Another accurate, rapid just as rapid as the infrared radiation devices. Measurement is direct and the methods simple. Some of these methods have the advantage of allowing the sample to be used for subsequent fat determination. The distillation with m-xylene or cumene (method 34) is not the most rapid method but it has the most well established accuracy.

OTHER METHODS

Refractometry of isopropanol extract of meat (method 35) proves to be a rapid (5-10 min.) and accurate method but the cost of the equipment approaches $2000. Another accurate, rapid and even more expensive method is gas-liquid chromatography (method 36) with a methanol extract of meat. The equipment costs about $18,000 though the USDA thinks that a $500 student model may be applicable.